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SEMICONDUCTOR OPTICAL DEVICES

The present invention relates to semiconductor optical devices, particularly but not solely for use in the field of bio-chemical or bio-medical analysis.

The use of optical techniques for the analysis of biological samples is a field of increasing importance, particularly in view of its potential for analysis at a molecular level. Various optical systems have been proposed hitherto: these systems have generally made use of a laser or other light emitter for directing light onto samples, typically successive samples in an array, and a separate photodetector for picking up light from the individual samples; typically the samples have been marked with a fluorescent dye, such that the incident light stimulates each sample to cause the emission of light of a different wavelength, which is picked up by the photodetector. Hitherto, such systems have been of large and complicated construction, for laboratory use.

We have now devised semiconductor optical devices which provide both light emission and light detection, for example enabling integration of an analytical system and providing a number of consequent advantages, but also of potential use in other applications: the devices may be of simple and small construction, enabling them to be used in the field.

In accordance with the present invention as seen from one aspect, there is provided a semiconductor emitter/detector optical device comprising a single substrate arranged for emitting light for incidence on a sample or other element and also responsive to light received from said sample or other element, the device further comprising means for monitoring a characteristic of the device which varies in dependence upon said light received from said sample or other element.

The device may include a photodetector, integrated on its substrate, for responding to the received light and providing an electrical output signal depending thereon. The photodetector may respond to received light of the same

wavelength as the light emitted by the device, for example reflected from a sample or other element, and/or it may respond to received light of a different wavelength, for example light emitted by a sample in response to stimulation by the light  
5 emitted by the device: such stimulated light emission may be produced by fluorescence.

Instead, the device may be such that the received light affects an electrical property of the device and so alters its current-voltage or impedance characteristic. In this case the  
10 monitoring means is arranged to monitor the current-voltage characteristic of the device, which varies depending on the received light (particularly the intensity thereof).

The device may comprise an array of light emitter/detectors integrated on the common substrate, these  
15 light emitter/detectors operating independently of each other and the monitoring means being arranged for monitoring the relevant device characteristic in respect of each emitter/detector, independently of the others. The device may then be used for performing an analytical test on a plurality  
20 of samples, one for each light emitter. The device preferably comprises a surface-emitting device, with an array of surface regions emitting light from the corresponding array of emitters, for the samples to be positioned in a similarly corresponding array over that surface. For example, a second  
25 substrate may be disposed over the device surface, this second substrate being formed with an array of recesses or chambers or flow ducts for receiving respective samples. The second substrate may be integrated with the device, or it may comprise a separate component. The plurality of light emitter/detectors  
30 may be arranged in a two-dimensional array, or in a linear array.

The device preferably comprises a resonant cavity light emitting device, for example a resonant cavity LED or a laser, preferably a vertical-cavity, surface emitting laser (or  
35 VCSEL).

A secondary optical cavity may be disposed over the emitting surface of the device, to form a coupled-cavity system, the secondary cavity including a chamber or flow duct for a sample.

5 In the case of a resonant cavity device, preferably a reflector thereof, through which the light output is emitted, comprises a plurality of alternating layers of high and low refractive index materials, and a layer of absorbing material is incorporated into or associated with the reflector. This  
10 absorbing layer serves to absorb light of a wavelength different from (typically longer than) the light emitted by the device: in absorbing light, electron-hole pairs are generated in the absorbing layer, so altering the current-voltage characteristic of the device. Because of its position at a  
15 node of the optical standing wave within the resonant cavity, the absorbing layer does not affect the light emitted by the device.

It will be appreciated that electron-hole pairs are produced in the absorbing layer, when light is absorbed in this  
20 layer. We have found that it is necessary for an electric field to exist across the absorbing layer, so that these electrons and holes are removed and effectively contribute to the current flow. In order to ensure this condition, preferably the absorbing layer is positioned in an undoped  
25 semiconductor region of the device: the PN-junction of the device then produces the desired electric field across the absorbing layer. The absorbing layer may be positioned in an undoped semiconductor layer or region lying between two groups of alternating high and low refractive index materials which  
30 form the reflector through which the light output of the device is emitted.

The device may typically comprise a two-terminal, vertically integrated device, with the monitoring means arranged to monitor the current-voltage characteristic of the  
35 device. For example, a constant voltage source may be

connected across the device and the monitoring means arranged to monitor the current flow: instead, the device may be fed from a constant current source and the monitoring means arranged to monitor the voltage across the device.

5           The monitoring means may comprise a circuit, part of which is integrated on the semiconductor substrate of the device.

          The semiconductor substrate may comprise a light-emitting layer disposed centrally between upper and lower  
10 reflectors, to form a resonant cavity, which is accordingly resonant at the wavelength of the emitted light. Instead, the cavity may be resonant at the wavelength of light to be detected, being a wavelength different from the emitted light: in this case, the layer, which is absorbing to the light to be  
15 detected, is disposed centrally between upper and lower reflectors, and a light emitting layer is disposed adjacent the upper and/or lower reflectors.

          The device in accordance with the present invention may comprise a light emitter element and a photodetector element,  
20 both integrated on a single substrate. The light emitter and/or photodetector may comprise a resonant cavity, in the form either of an LED or a laser. The photodetector may be arranged to detect light of a different wavelength from (e.g. of longer wavelength than) the light emitted by the light  
25 emitter: in this case, preferably the photodetector is arranged to respond preferentially to light of the former wavelength, so that it is relatively immune to any emitted light reflected or scattered back to the photodetector. For example, the photodetector element may be provided with a filter layer, at  
30 or adjacent its surface, which transmits light of the wavelength to be detected but blocks light of the emitted wavelength: this filter layer may be provided in an upper reflector of the photodetector.

          In one embodiment of device in accordance with the  
35 present invention, the semiconductor substrate is formed with

a resonant cavity between upper and lower reflectors, but a region of the upper reflector is removed to form an emitter, whilst another region of the substrate forms a photodetector and includes a layer, in the upper reflector, which filters out reflected emitted light.

Preferably a reverse bias applied to the photodetector places the diode close to its breakdown point: thus, in use, avalanche photo-detection occurs, thus substantially increasing the detection signal.

10 Devices in accordance with the present invention may comprise at least one pair of light emitter/detectors, only one directing emitted light onto a sample or other element and receiving reflected or return light from that sample or other element, whilst the other emitter/detector acts as a reference:  
15 the detection output signals of the two emitter/detectors are then combined together in a manner to cancel the noise components of these signals.

It will be appreciated that the above-described devices in accordance with the present invention may be miniaturised  
20 and mass produced, to provide mass-produced devices which are inexpensive yet reliable. Moreover, the samples may be positioned very closely to both the light emitter region and the light receiving region of the device, providing for high detection efficiency. It is particularly advantageous to  
25 provide for independent testing by means of an array of emitters/detectors on the same device.

In accordance with the present invention as seen from a second aspect, there is provided a device for the analysis or testing of a biological sample, the device comprising a  
30 single light emitter for directing light onto a sample, and a single photodetector for receiving light from the sample.

This device is accordingly a single-channel device for use with a single sample (or a single sample at a time). The device may be constructed to a very small size and  
35 inexpensively, so that it can be used with ease in the field,

and may indeed comprise a single use or disposable device.

The light emitter and photodetector may be mounted side-by-side and arranged for the biological sample to be positioned over them. The device may comprise a carrier  
5 substrate for the sample, positioned permanently or removably over the light emitter and photodetector.

Preferably the light emitter has a light emission peak at one wavelength and the photodetector has a light-absorbing peak at a different wavelength. In particular, the device may  
10 be arranged to detect fluorescent emission from the sample, stimulated by the light incident on it from the emitter.

The light emitter may comprise a vertical-cavity, surface emitting laser (VCSEL) or a resonant-cavity, light emitting diode (RCLED). The photodetector may comprise an  
15 identical device, having an emission peak at a different (longer) wavelength and used with a reverse bias, so as to act as a photodetector.

Embodiments of the present invention will now be described by way of examples only and with reference to the  
20 accompanying drawings, in which:

FIGURE 1 is a schematic view of a prior art system for the optical analysis of biological samples;

FIGURE 2 is a diagrammatic section through a semiconductor emitter/detector device forming a first  
25 embodiment of the present invention;

FIGURE 3 is a section, on enlarged scale, of part of the device of Figure 1, showing the optical intensity variation therein;

FIGURE 4 and 5 are graphs showing calculated electrical  
30 field profiles in the reflector which incorporates an absorbing layer;

FIGURE 6 is a graph showing the light emission/detection spectra of this device;

FIGURE 7 is a diagrammatic section through a  
35 semiconductor emitter/detector device forming a second

embodiment of the present invention;

FIGURE 8 is a view of the device of Figure 7;

FIGURE 9 is a diagrammatic section through a semiconductor emitter/detector device forming a third  
5 embodiment of the present invention;

FIGURE 10 is a diagrammatic section through a further embodiment of semiconductor emitter/detector device in accordance with the present invention;

FIGURE 11 is a diagrammatic section through a  
10 modification to the device of Figure 2;

FIGURE 12 is a schematic cross-section through a yet further embodiment of device in accordance with the invention;

FIGURE 13 is a graph showing the emission spectra of two light emitters used in the device in Figure 12; and

15 FIGURE 14 is a plot showing the detection performance of one of the light emitters of the device of Figure 12, used with reverse bias to act as a photodetector.

Referring to Figure 1, there is shown a prior art system for the optical analysis of an array of biological  
20 samples, each of the samples being marked with a fluorescent dye. The system comprises a tray T the upper surface of which is formed with a two-dimensional array of recesses or wells R which receive respective samples. A laser or other source S is provided to direct light onto the samples: means are  
25 provided for scanning the light beam onto the samples in succession. A photodetector P is provided to pick up the light emitted, by fluorescence, from the successive samples, the output of the photodetector being passed to a processing unit.

Referring to Figure 2, there is shown a semiconductor  
30 device in accordance with the present invention in the form of a vertical-cavity, surface-emitting laser (or VCSEL), for use in performing an analytical test on a biological sample. The device comprises a semiconductor substrate having cavity layers 10, 12 and an intermediate gain material layer 14, and upper and  
35 lower multi-layer reflectors 16, 18: each reflector comprises

a plurality of alternating layers of different materials, respectively of high and low refractive index, each layer being a  $\frac{1}{4}$  wavelength thick. The device is provided with electrodes 20,22 on its top and bottom surfaces: in use, a voltage is applied between the upper and lower electrodes 20,22 to provide a current flow through the device; this current excites the device to cause lasing within its resonant cavity, the laser output light O being emitted through the top surface of the device.

10 In accordance with the present invention, the laser output is directed onto a biological or bio-chemical sample (not shown) positioned on or above the device, the sample being marked with a fluorescent dye. The laser output O stimulates the sample, causing the emission of light of a longer wavelength than the laser output light: some of the light D emitted from the sample returns and passes into the device.

The upper reflector 16 includes a layer 24 of narrow bandgap material, forming an absorbing layer for the light of the wavelength emitted by the sample. The absorbing layer 24 is disposed at a position which corresponds with a node in the internal optical standing wave of the device cavity: the variations in optical intensity are shown by the trace W in Figure 3 and show the absorbing layer 24 at a node; accordingly, the absorbing layer 24 does not absorb the light emitted by the device or affect its lasing characteristics.

In use, the light D picked up by the device, from the sample, is absorbed by the absorbing layer 24, generating hole-electron pairs, which accordingly alter the current-voltage characteristic of the device. A constant voltage source may be connected across the device, and means provided to monitor the current flow, the current varying in dependence on the intensity of light D received from the sample. Alternatively, the device may be fed from a constant current source, and means provided to monitor the voltage across the device, which again varies in dependence upon the intensity of light D received



from the sample.

Referring in more detail to the structure of the upper reflector 16, in a preferred form this comprises a first group of alternating layers of high and low refractive index materials, then an undoped layer of semiconductor material, then a second group of alternating layers of high and low refractive index materials, with the absorbing layer disposed in the undoped layer between the two groups of alternating high and low refractive index layers. Figures 4 and 5 show the two groups of alternating high and low refractive index layers at 16a, 16b and the absorbing layer at 24, the distance being measured outwardly from the optical cavity of the device: the trace E shows the calculated electrical field. For the emitted light wavelength of 645 nm, the absorbing layer 24 is at a node or minimum in the electrical field and does not therefore affect the light being emitted by the device: for the returned light of 675 nm wavelength, this is no longer the case and the absorbing layer 24 serves to absorb this light.

Figure 6 shows the emission and detection spectra of a device which we have produced in accordance with the above teachings and shows clearly that the emitted light is unaffected by the absorbing layer, whilst effective detection of light occurs at longer wavelengths, extending up to 890 nm.

By providing the absorbing layer in an undoped region of semiconductor of the device, it is ensured that an electric field exists across the absorbing layer, so that the electron and hole pairs (which are generated upon absorbing the longer-wavelength light) are effectively removed and contribute to the current flow through the device. In this way, an external signal is provided, dependent on the intensity of the received light of longer wavelength.

In a modification of the device of Figure 2, the device may comprise a resonant cavity LED instead of a VCSEL.

Referring to Figures 7 and 8, there is shown a semiconductor device in the form of a two-dimensional array

of vertical-cavity, surface-emitting lasers L, for use in performing an analytical test on a corresponding array of biological or bio-chemical samples B. The device comprises a semiconductor substrate having cavity layers 10,12 and an intermediate gain material layer 14, and upper and lower multi-layer reflectors 16,18: each reflector comprises a plurality of alternating layers of high and low refractive index materials, each layer being a  $\frac{1}{4}$  wavelength thick. The device is provided with an array of electrodes 20 on its upper surface and with a common or ground electrode 22 on its bottom surface. In use, a voltage is applied between each upper electrode 20 and the bottom electrode 22, to provide respective current paths through the device. In each current path, the device is excited to cause local lasing action within the device cavity, the laser output light O being emitted through the spaces or windows 20a between the electrodes 20 on the top surface of the device. It will be appreciated that the arrangement forms a two-dimensional array of lasers.

An interface layer 26 is formed over the upper electrode 20 and upper reflector 16, and a sample-receiving substrate 28 is disposed over the interface layer 26. The upper surface of the sample-receiving substrate 28 is formed with a two-dimensional array of recesses or wells 29, for receiving respective samples B: the wells 29 are aligned with respective windows 20a between the upper electrodes 20 of the device; thus, there is one independent laser for each sample well 29. The sample-receiving substrate 28 may be integrated with the semiconductor device: alternatively, it may comprise a separate component, as shown in Figure 8, which can be removed and disposed off if desired.

The upper reflector 16 of the device of Figure 7 includes a layer 24 of narrow bandgap material, forming an absorbing layer for light returned by each sample. The absorbing layer 24 is positioned, within the reflector 16, in the same manner as described with reference to Figures 2 to

6.

Light O emitted from each laser is incident on the corresponding sample B, which is marked with a fluorescent dye, thus stimulating the sample to cause emission of light of longer wavelength. Some of this light returns to the respective laser L of the device and is absorbed locally by the absorbing layer 24, with the result of altering the current-voltage characteristic of the respective laser L. A constant voltage source may be connected across each laser L, via its respective upper electrode 20 and the ground electrode 22, and means provided for monitoring the current flow through that laser: alternatively each laser L may be fed from a constant current source, and means provided for monitoring the voltage across that laser.

15 In a modified form of the embodiment shown in Figure 7, each laser L may be provided with a photodetector, integrated within the semiconductor device, to receive light D returned from the respective sample B, the absorbing layer 24 being dispensed with. The photodetector of each laser will detect laser light reflected back from the respective sample, or light emitted from the sample in response to stimulation by the laser light. The outputs of the photodetectors are connected to a monitoring circuit.

It will be appreciated that the device of Figure 2, or the above-described modified form thereof, may comprise an array of resonant cavity LEDs, instead of an array of VCSELs.

In another modified form of the embodiment shown in Figure 7, again with the absorbing layer 24 dispensed with, the output light from each laser is reflected by the respective sample and returns into the laser, acting to modify the current-voltage characteristic of the laser. A circuit is connected to the electrodes 20 of the lasers, to monitor the current through and/or voltage across each laser.

Referring to Figure 9, there is shown a semiconductor device in the form of a vertical cavity, surface-emitting

laser, having a second optical cavity 30 in contact with it, the cavity containing a biological or biochemical sample or including a flow duct for such a sample. The device comprises a semiconductor substrate having cavity layers 10,12 and an intermediate gain material layer 14, and upper and lower multi-layer reflectors 16,18: each reflector comprises a plurality of alternating layers of different materials, respectively of high and low refractive index, each layer being a  $\frac{1}{4}$  wavelength thick. The device is provided with electrodes 20,22 on its top and bottom surfaces: in use, a voltage is applied between these electrodes to provide a current flow through the device, exciting the device to cause lasing within the device cavity and emission of laser output light O through the top surface of the device.

15. The second optical cavity 30 comprises a container 32 for the sample B, the upper side of the container 32 comprising a multi-layer reflector 34 of construction corresponding to the reflectors 16,18 of the laser device. A transparent spacer 36 is interposed between the underside of the container 32 and the upper surface of the device.

The two optical cavities form a coupled-cavity system, the emitted laser light O being transmitted through the sample B, then reflected, by the reflector 34 of the second cavity, back into the laser device. This return or feedback light D is amplified within the laser device and so modifies its current-voltage characteristic. A circuit is connected to the electrode 20 of the laser, to monitor the current through and/or voltage across the laser.

In a modified form of the embodiment of Figure 9, the reflector 16 of the device includes a narrow bandgap absorbing layer, in the same manner as described with reference to Figures 2 to 6, for absorbing stimulated-emission light from the sample B, of a different wavelength to the laser output. In this form, the device may comprise a resonant cavity LED, instead of a VCSEL.

As mentioned previously, a semiconductor device in accordance with the present invention may comprise a light emitter element and a photodetector element, both integrated on a single substrate. Either the light emitter or  
5 photodetector, or both, may comprise a resonant cavity, in the form either of an LED or a laser. Where the photodetector is intended to detect light of a different wavelength from the light emitted by the light emitter, then it may be desirable for the photodetector to be arranged to respond preferentially  
10 to light of the former wavelength, so that it is relatively immune to some of the emitted light being returned, by scattering, to the photodetector. For example, the photodetector element may be provided with a filter layer, at or adjacent its surface, which is relatively opaque to light  
15 of the wavelength of the light emitted by the light emitter element of the device, but relatively transparent to light of the wavelength to be detected.

Figure 10 shows a device in which the upper reflector of the photodetector element includes a layer 38 forming an  
20 absorption filter blocking any reflected emission light. The device comprises a semiconductor substrate having cavity layers 10, 12 and an intermediate gain material layer 14, and upper and lower multi-layer reflectors 16, 18 each comprising a plurality of alternating layers of high and low refractive  
25 index materials, as devised above with reference to Figure 2, for example. The upper reflector 16 has, however, been etched away over one portion of the substrate, and a trench 40 separates the two portions of the device, to form emitter and photodetector elements. The device is provided with electrodes  
30 20a, 22a and 20b, 22b on the top and bottom surfaces of the emitter and photodetector elements of the device, a forward bias being applied across electrodes 20a, 22a and a reverse bias being applied across electrodes 20b, 22b. In use of the device, light 0 of a first wavelength is emitted from the upper  
35 surface of the emitter element of the device: any reflected

light of this wavelength, incident on the photodetector element, is filtered out by the absorbing layer 38, whilst light of e.g. longer wavelength passes through this layer and is detected within the photodetector element. As described previously, a constant-voltage source may be connected across the photodetector electrodes 20b, 22b and a circuit provided to monitor the current, or instead a constant-current source is connected across the electrodes 20b, 22b and a circuit provided to monitor the voltage between them.

Conveniently, the trench 40 may be annular in form, encircling a central emitter element of relatively small diameter (perhaps of the order of 10 microns), and itself encircled by a photodetector element of relatively large diameter (perhaps of the order of 500 microns).

Preferably the reverse bias applied to the photodetector element places this diode close to its breakdown point, so that, in use, avalanche photo-detection occurs: a substantially increased detection signal (of the order of a thousand-fold increase) can accordingly be achieved.

It will be noted that, in the device of Figure 2 for example, the light-emitting layer 14 is disposed centrally between the upper and lower reflectors and the cavity is resonant at the wavelength of the emitted light. Instead, and referring to Figure 11, the cavity may be resonant at the wavelength of light to be detected, in which case the layer 24, which is absorbing to the light to be detected, is disposed centrally between the upper and lower reflectors 16,18: further, the emitting layer 14 is disposed adjacent either the upper or lower reflector.

Whilst in the embodiment of Figures 7 and 8, for example, the substrate comprises a plurality of light emitters arranged in a two-dimensional array, these may instead be arranged in a linear array, for example adjacent a passage for the flow of fluid, arranged to examine the fluid at successive points along its flow path.

It will be appreciated that, in each of the above-described embodiments, the monitoring means may comprise a circuit part of which is integrated on the semiconductor substrate of the device.

5 Referring to Figure 12 of the drawings, there is shown a device for the analysis of a biological or other sample, the device comprising a semiconductor light emitter 50 and a semiconductor photodetector 52 mounted side-by-side on a substrate 54. A sample-carrier 56 is positioned over the light  
10 emitter 50 and photodetector 52 and comprises a transparent substrate the upper surface of which is formed with a recess or well to receive the sample S to be analysed or tested. The substrate 56 may form a permanent part of the device, or it may be removable and replaceable.

15 The device is arranged so that the light emitted by the emitter 50 is incident on the sample S and the photodetector 52 picks up light returned from the sample S. In the sample shown in Figure 12, the light emitter 50 comprises a resonant-cavity, light emitting diode (RCLD) with an emission peak at  
20 a wavelength of 650nm: the photodetector comprises an identical device except that it has an emission peak at 670nm and is used with a reverse voltage bias (so as to act as a photodetector rather than light emitter). In use of the device, the sample S is marked with a fluorescent dye: accordingly, the light  
25 of 650nm wavelength which is incident on the sample, from the emitter 50, stimulates the sample to cause emission of light of the longer-wavelength, which is picked up by the photodetector 52.

We have made and tested light emitters and  
30 photodetectors in the form of GaInP crystals having multiple-layer Bragg reflectors to produce circular beams perpendicular to their surfaces. By control of the thickness of these crystals, we formed two devices with emission peaks at 650nm and 670nm, respectively: the first such device is used as the  
35 emitter 50 and the second device, under reverse bias, as the

photodetector 52.

Figure 13 shows the emission spectra of the two devices, one having a peak at 650nm and the other a peak at 670nm.

5        Figure 14 shows the detected photovoltage, of the photodetector 52, in response to an optical signal. We found detection possible down to a noise floor of 3nW: for a typical fluorescent dye with a radiative lifetime of 5ns, this corresponds to the detection of the fluorescent emission from  
10 just 51 molecules, demonstrating the extremely high sensitivity of the photodetector.

It will be appreciated that the device described with reference to Figure 12 also comprises a battery power source for applying forward and reverse voltages to the emitter 50 and  
15 photodetector 52, respectively: the device further comprises a circuit for measuring the voltage or current output of the photodetector.